

## NISTTech

METHOD AND APPARATUS FOR PYROLYSIS PLOT-CRYOADSORPTION HEADSPACE SAMPLING AND ANALYSIS [NIST Docket No. 12-038]

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### Abstract

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Headspace analysis is a technique in which a gas that has previously been in contact with a condensed phase (solid or liquid) is examined for the presence of volatile compounds released into the gas. The partitioning of analytes into the gas phase can be understood or even predicted with thermodynamic considerations. This thermodynamic relationship can sometimes make possible the approximation of analyte concentration in the original condensed phase. Headspace analysis is most easily accomplished for a volatile analyte, in a nonpolar matrix. Other sample presentations become progressively more difficult. Sampling methods from headspaces can be either static or dynamic. In static methods, one typically pressurizes a sealed vial or vessel containing the condensed analyte (to slightly above atmospheric pressure), then sampling is done of the pressurized headspace through a septum. Sampling can be done with a gas tight syringe (with or without a syringe valve), a multiport sampling valve, or with a solid phase microextraction (SPME) fiber. In dynamic methods, a flow of carrier or sweep gas is applied to the matrix containing the analyte. The stream is then collected in a cryostat, absorbent or solvent, thus this method is often referred to as purge and trap. The sweep gas can be under a positive pressure or drawn through the sample at reduced pressure; either approach has its advantages and pitfalls. When the analyte in the headspace gas is at a trace level, or when an exhaustive analysis of all constituents is desired, purge and trap methods are often preferred over static headspace or even modern SPME approaches. For analytes of very low volatility, longer collection times are required to collect sufficient sample for analysis. Moreover, static and SPME methods are often difficult to make quantitative and reproducible. These techniques are often more suited to survey analysis, or to verify the presence of an analyte. They are often not suitable for temperature dependent studies that are critical in the validation of other analytical methods

### Inventors

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- Bruno, Thomas J.

### References

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- 12-038Application

### Status of Availability

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